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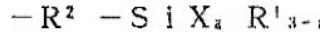
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(54) ROOM-TEMPERATURE-CURABLE COMPOSITION

(57)Abstract:

PURPOSE: To obtain a room-temp.-curable compsn. which exhibits a sufficient depth cure rate and is free from troubles, such as the viscosity increase of the main component, even during long storage by compounding a specific org. polymer with a cure catalyst, an org. amine compd., and a filler.

CONSTITUTION: This compsn. is prep'd. by mixing and kneading 100 pts.wt. org. polymer which is derived from a hydroxylated polyoxalkylene polymer obtd. by polymerizing an alkylene oxide using an initiator in the presence of a composite metal cyanide complex as the catalyst and has hydrolyzable silicon groups represented the formula (R1 is a 1-20C monovalent hydrocarbon group, R2 is a divalent org. group; X is a hydroxyl or hydrolyzable group; and a is 1-3) and a total content of ionic impurities of 50ppm or lower with 0.01-10 pts.wt. cure catalyst selected from among 20C or lower aliph. carboxylates of divalent tin, divalent bismuth, and divalent lead, 0.01-10 pts.wt. org. amine compd., 50-250wt.% (based on the polymer) filler, and optional plasticizers and additives.



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CLAIMS

[Claim(s)]

[Claim 1]A curing catalyst (B) chosen from the following organic polymer (A), carboxylates of divalent tin, carbonylates of divalent bismuth, and carbonylates of divalent lead, and an organic amine compound (C) and a room-temperature-curing nature constituent containing a bulking agent (D). [Organic polymer (A) is derived from a hydroxyl group content polyacrylonitrile polymer (F) produced by polymerizing acrylonitrile oxide in an initiator by making a dicyanopropyl metal cyanide complex (E) into a catalyst. An organo polymer (A) whose total amount of an ionic impurity it has a hydrolytic silicon group expressed with a formula (1), and is 50 pm or less.



As for R₁, a hydroxyl group or a hydroxyl group basis, and a of substitution of this carbon numbers 1–20 or an unsubstituted univalent hydrocarbon group, and R₂² are the integers of 1–3 & a divalent organic group and X₁ among a formula.

[Claim 2]A room-temperature-curing nature constituents of Claim 1 whose organic amine compound

(C) is with a carbon number of 20 or less amine.

[Claim 3]Claim 1 or 2 room-temperature-curing nature constituents which are a compound in which an organic amine compound (C) has a hydrolytic silicon group expressed with an amino group and a formula (2) to intramolecular.



As for a divalent organic group and R₁¹ a hydroxyl group or a hydroxyl basis, and b of substitution of the carbon numbers 1–20 or an unsubstituted univalent hydrocarbon group, and X₁ are [R₂²] the integers of 1–3 amines a formula.

[Claim 4]A room-temperature-curing nature constituent of either of the Claims 1–3 which is an ionic impurity in which an ionic impurity contains metal cyanide compounds and/or an alkali metal compound resulting from a composite metal cyanide complex (E).

[Claim 5]An organic polymer (A) an ionic impurity contained in a polymer (F). After an ionic impurity contained in a polymer (F) by removing this salt from a polymer (F) after considering it to be a polymer (F) as an insoluble salt intrinsically shall be 50 pm or less. A room-temperature-curing nature constituent of either of the Claims 1–4 which is an organic polymer produced by introducing a hydrolytic silicon group into a polymer (F).

[Claim 6]An organic polymer (A) an ionic impurity contained in a terminal unsaturated group introduction thing (G) of a polymer (F). After an ionic impurity contained in a terminal unsaturated group introduction thing (G) by removing this salt from a terminal unsaturated group introduction thing (G) after considering it to be a terminal unsaturated group introduction thing (G) as an insoluble salt intrinsically shall be 50 pm or less. A room-temperature-curing nature constituent of either of the Claims 1–4 which is an organic polymer obtained by making it react to a silicon hydride compound expressed with a terminal unsaturated group introduction thing (G) and a formula (3).



A hydroxyl group or a hydroxyl basis, and a of R₁¹ are the integers of 1–3 among a formula substitution of the carbon numbers 1–20 or an unsubstituted univalent hydrocarbon group, and X.

[Claim 7]A room-temperature-curing nature constituent of either of the Claims 1–6 whera hydroxyl

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

Industrial Application[This invention relates to the room-temperature-curing nature constituent hardened under hygroscopic-moisture existence.

[0001] Description of the Prior Art[The method of using it for a sealing material, adhesives, etc., using the hardening reaction of various kinds of organic polymers which have a hydrolytic silicon group at the end which is known as conventional, for example, modified silicone, system resin is known well, and is a useful method industrially.

[0002] [Problems] to be Solved by the Invention[The organic polymer which has a hydrolytic silicon group at such the end is proposed by JP-45-343193B, JP-61-156252D, etc., for example.

[0003] [Description of the Invention] This invention relates to the room-temperature-curing nature of an organic polymer which has a hydrolytic silicon group among the organic polymers which have a hydrolytic silicon group, using what is called a curing catalyst. As such a curing catalyst is usually performed, such as metal salt of carboxylic acid, acidity, or a basic compound of carbon, and carbonyl of tin and other organotin compounds are especially common.

[0004] To the organic polymer which has a hydrolytic silicon group at the end, various bulk agents, a plasticizer, 1,4-dibutyl-2,6-naphthoquinone, called what is called a 1 liquid type, and a 2 liquid type as a gestant of the combination when using the main composition of the room-temperature-curing nature which includes an adhesive graft agent, a curing catalyst, various stabilizer, etc., for a sealing material, adhesives, etc.

[0005] It is known as a 2 liquid type among those is divided into base resin and a hardening agent, it mixes solvents, etc., with two ingredients if needed further, constructs, and makes it harden in a service space at the time of use. As for the hardening agent in the 2 liquid type, it is common to include the curing catalyst, and as the curing catalyst, Since a hardened material shows good compression restoration nature to an elasticity, leaden carboxylate or carboxylate of diisobutylene, and the organic amine compound, and the method of specifically using octyltin acid and laurylamine are proposed (ref. to JP-61-08867B).

[0006] However, in a 2 liquid type, as an organic polymer which has an end hydrolytic silicon group, after a silanol compound's trying the polyester compound of the comparatively short molecular weight proposed by the aforementioned well-known example, setting it and carrying out Polymer Division quaternization. If octyltin acid and lauryl amine are used as a curing catalyst when the polymer and building agent which have a hydrolytic silicon group are used as base resin, by a part far from the surface of a cure rate, especially a cured body, the so-called cure rate of the depths is insufficient, and it cannot be satisfied.

[0007] When the organic polymer which manufactured as a catalyst, the composite metal cyanide from the polyoxyethylene polymer which has a hydrolytic silicon group] in JP-3-43448A and JP-3-7682A, is used as one resin, in order not to usually perform strict mutual contact that is, let the amount of [In base resin] water content 100 when manufacturing base resin although the hardenability of the depths improves remarkably if this curing catalyst is used, there was a case

where the viscosity of base resin rose during long-term preservation, and use top inconvenience arose.

[Means for Solving the Problem] This invention is the following invention which is going to cancel such a fault.

[0010] A curing catalyst (B) chosen from the following organic polymer (A), carboxylate of diivalent tin, carboxylate of diivalent bismuth, and carboxylate of diivalent lead, and an organic amine compound (C) and a room-temperature-curing nature constituent containing a bulking agent (D).

[0011] Organic polymer] It is derived from a hydroxyl group content polyoxyethylene polymer (F) produced by polymerizing alkylene oxide in an earlier by making a composite metal cyanide complex (E) into a catalyst. An organic polymer (A) shows total a hydrolytic silicon group expressed with a formula (1), and is 50 ppm or less.



[0013] for R¹, a hydroxy group or a hydrolytic basis, and a of substitution of the carbon numbers 1-2 or an unsubstituted univalent hydrocarbon group, and R² are the integers of 1-3 a divalent organic group and X amine formula.

[0014] [Organic polymer] An organic polymer (A) derived from a hydroxyl group content polyoxyethylene polymer (F) produced by polymerizing alkylene oxide in an initiator by making a composite metal cyanide complex (E) into a catalyst.

[0015] Using a composite metal cyanide complex (E) using the conventional alkaline metal catalyst, N_nM_n is rather than J and M, a hydroxyl group content polyoxyethylene polymer (F) of hydroxylsity can be obtained more in the amount of Polymer Division.

[0016] A complex which uses zinc hexacyanocobaltate as the main ingredients as a composite metal cyanide complex (E) is preferred, and the ether and/or especially an alcohols complex are preferred. The presentation can use what is intrinsically indicated to JP-S6-22720/B. As either, ethylenglycol dimethyl ether (dgme), diethylethanol dimethyl ether (fia line), etc., are preferred, and especially pentanetriol, diethyl, a shikoku airin, and these. One sort singular use or two or more sorts of concomitant use may be sufficient as these. As alcohol, t-butanol is preferred.

[0017] A compound which has 2-10 active hydrogen as an initiator is preferred, a polyhydroxy compound is preferred, and a polyhydroxy compound which has 2-4 hydroxy groups especially is preferred -8 ples. Specifically Ethylene glycol, diethylene glycol, propylene glycol, dipropylene glycol, neopentyl glycol, 1,4-butanediol). There is poly of low molecular weight from an object produced by making alkylene oxide react to 1,6-hexamethylene diol, glycerin, triethylene glycol, pentanetriol, diethyl, a shikoku airin, and these. One sort singular use or two or more sorts of concomitant use like a alcohol can also be used. An unsaturation group content mono- hydroxy compound like a alcohol is mentioned as the reducing method. Methods of using it,

[0018] A total amount of an ionic impurity of an organic polymer (A) of this invention is 50 ppm or less. This invention when especially in salt form in an ionic impurity containing metallic compounds and/or an alkali metal compound resulting from composite metal cyanide complex (E). It is preferable that ionic impurities are 30 ppm or less and 20 more ppm or less.

[0019] By enduring such metal impurity quantity, the storage stability of an organic polymer (A) and a hardenability constituent of this invention is improved more, and its hardenability which was excellent in order not to have an operation of a curing catalyst is acquired.

[0020] Following, (I) is a method of (III) are mentioned as the reducing method. Methods of using it, when removing metallic compounds resulting from especially a composite metal cyanide complex (E) include (II). Especially since a method can reduce an ionic impurity effectively and economically, it is desirable.

[0021] How to remove this salt from a polymer after making into an recyclable salt, intrinsically an ionic impurity contained in a polymer to a polymer. After adding the Nonion system surface-active agent (a compound, water, and it needed) which specifically react to an ionic impurity and can form an insoluble salt to a polymer intrinsically, by drying, a salt is deposited, and there is a method of next removing this salt. As a compound which can form a salt, chloride, sulfate salt, nitro acid.

phosphoric acid, pyrophoric acid, disodium diphosphoric pyrophosphate, etc. are preferred. A deposited salt is removable by filtration operation, adsorbing operation, etc.

[0022] (I) is a method of removing the salt from a polymer after making into an recyclable salt, intrinsically an ionic impurity contained in a polymer to a polymer. After adding the Nonion system surface-active agent (a compound, water, and it needed) which specifically react to an ionic impurity and can form an insoluble salt to a polymer intrinsically, by drying, a salt is deposited, and there is a method of next removing this salt. As a compound which can form a salt, chloride, sulfate salt, nitro acid.

phosphoric acid, pyrophoric acid, disodium diphosphoric pyrophosphate, etc. are preferred. A deposited salt is removable by filtration operation, adsorbing operation, etc.

[0022] How to make an anion exchange resin and/or cation exchange resin contact, and to remove an ionic impurity after adding a solvent to a polymer.

[0023] To a buffer pH and arbitration, by ammonia and a chelating agent (II). After proceeding, A method of removing metallic compounds which originate in a composite metal cyanide complex (E) after adding a method and fatty alcohol from which metallic compounds resulting from a composite metal cyanide complex (E) are removed, and a chelating agent, a method of removing metallic compounds which originate in a composite metal cyanide complex (E), after proceeding with an oxidizer.

[0024] With an ionic impurity as used in the field of this invention, originate in a composite metal cyanide complex (E). A carbon and a silicon, such as 2-aminobutyl, cobalt cyanide ion, and a silane ion. Mix as an impurity in a process manufacturing an organic polymer (A). Sodium ion, Ni²⁺ metal ion like potassium ion, carboxylate on that polyoxyethylene generated in response to oxidation at a process of manufacturing a halogen ion, organically polymer (A); An organic polymer (A) at a process to manufacture An ester bond. All the anions, such as catalysts metal salt added when making carbonate combination etc. form, and cations are included.

[0025] As for an organic polymer (F), what replaced a hydrogen atom in a hydroxyl group of a hydroxyl group content, polyoxyethylene glycol monolynly ether, can also be used.

[0026] As for the number of hydroxyl groups per molecule of a polyoxyethylene polymer (F) used for this invention, it is preferred that they are 2-10 pieces. It is especially referred from balance of physical properties, such as viscosity, fineness, and elongation, that they are especially 2-4 pieces 2-8 pieces.

[0027] Desirable polymers (F) are polyoxypropylene diol, polyoxypropylene triol, and polyoxypropylene tetraol. When using for a method of the following (b) or (e), unsaturation group and polyoxyethylene monoacids, such as polyoxypropylene glycol monolynly ether, can also be used.

[0028] As for a hydroxyl value conversion molecular weight, of a polyoxyethylene polymer (F), 5000-30000 are preferred, and 8000-30000 are more preferred.

[0029] A hydroxyl value conversion molecular weight of this invention says a thing of a molecular weight calculated of a product of a functional group number of an initiator, and a molecular weight per hydroxyl group of polyoxyethylene of a polymer used when manufacturing a polyoxyethylene polymer (F) containing terminal hydroxyl groups.

[0030] An organic polymer (A) has a hydroxyl silicon group expressed with a formula (1).



[0032] For R₁, a hydroxyl group or a hydroxide basis, and a of substitution of the carbon numbers 1-20 or an unsaturated univalent hydrocarbon group, and R₂ are the integers of 1-3 a divalent organic group and X amino a formula.

[0033] R in a formula (1) is a univalent hydrocarbon group substitution of the carbon numbers 1-20, or unsubstituted, and are with a carbon number of eight or less alkyl propyl group, a phenyl group, and a halogen group, a cyclohexyl group, a phenyl group, etc. especially preferable.

[0034] X is a hydroxyl group or a hydroxide basis, and is a halogen atom, an alkoxy group, an aryloxy group, an amido group, an amino group, a KETOKSHi male group, and a hydroxide basis group as a hydroxide basis, for example. As for especially a carbon number of a hydroxide basis which has a carbon atom among these, four or less or six or less is preferred. As desirable X, with a carbon number of four or less lower alkoxy group especially a methoxy group, an ethoxy basis, a propoxy group, etc. can be illustrated, is a number of 1-3 and 2 or 3 is preferred.

[0035] A, a manufacturing method of an organic polymer (A) is explained. The organic polymer (A) in this invention can introduce a hydroxyl group into an end of a hydroxyl group content polyoxyethylene polymer (F) by a method which is stated to the following (**) - (**) Such a compound is liquefied at a room temperature, and when a cured body holds uses for a sealing material, adhesives, etc. also at low temperature comparatively, it is provided with the desirable characteristic.

[0036] (*) A method to which a silicon hydroxide compound expressed with a terminal unsaturated group introduction thing (G) and a formula (3) of a polymer (F) is made to react.

[0037]HRSX₁R₁¹-R₂¹...-(3)

[0038]However, R¹ formula X, and a are the same as the above.

[0039]A method of obtaining, terminal unsaturated group introduction thing (G) of a polymer (F), After setting terminal hydroxyl groups On of a polymer (F) to OM (M is an alkaline metal), there is a method of making a compound which has a functional group which can react to a method or an unsaturation group, and a hydroxyl group which a made to react to unsaturation group content halogenated hydrocarbon, such as an allyl chloride, react, to a polymer (F), and combining by ester bond, a methylene bond, carbonyl combination, etc. When polymerizing alkylene oxide or mono-hydroxy compound, a methylene bond, carbonyl combination, such as allyl glycidyl ether, by an initiator by carrying out copolymerization of the unsaturation group content alkylene oxide, such as allyl glycidyl ether.

[0040](**) How to make a polymer (F) react to a compound which has a hydroxyl silicon group expressed with an isocyanate group and a formula (1).

[0041](**) A method to which W basis of a silicon compound expressed with a formula (4) to this isocyanate group is made to react after making polyisocyanate compounds, such as tolylene disocyanate, group is made to react after making polyisocyanate compounds, such as tolylene disocyanate group is made to react after making polyisocyanate compounds, such as allyl glycidyl ether.

[0042]R₁¹-SiX₁-R₂¹-...-(4)

[0043] —X, and a — the above — the same — R⁵ are a divalent organic group and the active hydrogen containing group as which W was chosen from a hydroxyl group, a carboxyl group, a sulfhydryl group, and an amino group (the 1st class or the 2nd class).

[0044](**) A method to which an unsaturation group introduction thing (G) of a silicon compound expressed with a formula (4) to a polymer (F) by removing this salt from a polymer (F) after an ionic impurity contained in a polymer (F) by removing this salt from a polymer (F) shall be 50 ppm or less, a hydroxyl silicon group is introduced to a polymer (F), and it is considered as an organic polymer (A).

[0045]A, a hydroxyl group contained in a terminal unsaturated group introduction thing (G) by removing this salt from a terminal unsaturated group introduction thing (G) after considering it in a terminal unsaturated group introduction thing (G) as an insoluble salt intrinsically shall be 50 ppm or less. It is made to react to a silicon hydroxide compound expressed with a terminal unsaturated group introduction thing (G) and a formula (2), and is considered as an organic polymer (A).

[0046]A molecular weight of an organic polymer (A) in this invention is computed polymer (F) by removing this salt from a polymer (F) which is a raw material. As for this value conversion value molecular weight of polymer (F) is 5000-30000 is preferred. If elongation will become hard boy in a body if lower than 5000, and 30000 is exceeded, pliability and elongation of a cured body are satisfactory, but viscosity of the polymer itself [this] becomes a remarkably high, and practicability becomes low. Especially 8000-30000 are preferred.

[0049]In catalyst A, a curing catalyst, (B), and an organic amine compound (C), which are chosen from carbonyls of diphenyl ether, carbonyls of phenol bismuth, and carbonyls of divalent metal in this invention are used. Although these metal carbonyls itself acts to promote hardening of an organic polymer (A), it is insufficient for the purpose of this invention, and it is indispensable to use together with an organic amine compound (C).

[0050]A, for a curing catalyst (B), is preferred that it is a salt of with a carbon number of 20 or less of straight chain shape or a letter of branching aliphatic carboxylic acid. As with a carbon number of 20 or less aliphatic carboxylic acid, there is a heptanoic acid, octanoic acid, decanoic acid, lauric acid, stearic acid, etc. A curing catalyst (B) may be a mixture.

[0051]Especially, the amount of these metal carboxylic acid used has 0.01 - 3 preferred weight section 0.01 to 10 weight section to organic polymer (A) 100 weight section.

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例	35	36	37	38	39	40
主 斧	M7	M8	M9	M10	M11	M12
硬化剤	K1	K1	K1	K2	K3	K4
軸入部	0. 6	0. 6	0. 4	0. 7	0. 5	0. 4

[Table 7]

例	41	42	43	44	45
主 斧	M7	M7	M1.4	M1	M2
硬化剤	K1	K1	K1	K5	K5
軸入部	1. 5	1. 5	1. 5	2. 5	2. 5

[Table 8]

記述例	含有する金属カルボン酸盐
K.1	カクテル醸造スズ (2面)
K.2	(日本酒度数ニッカオカタックスズ) オカタックスズ (2面) (清酒度数ニカタックスズ 8.3)
K.3	バーサチャーフェビスズ (2面)
K.4	(The Standard Chalice) 社用脚 (SWITE VERSALITH) ネオガラフスズ (2面)
K.5	ダブルスコットシャーレ (4面)

[0088] [Effect of the Invention]Storage stability is improved remarkably and the hardenability constituent of this invention has this good hardenability of the depths.

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CORRECTION OR AMENDMENT

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[Written Amendment]

[Document to be Amended]

[Description]

[Item(s) to be Amended]

[Claim(s)]

[Method of Amendment]

[Proposed Amendment]

[Claims]

[Claim 1A] curing catalyst, (B) chosen from the following organic polymer (A), carboxylates of divalent tin, carboxylates of divalent bismuth, and carboxylates of bivalent lead, and an organic amine compound (C) and a room-temperature-curing nature constituent containing a bulking agent (D). (Organic polymer) is derived from a hydroxyl group content polyisobutylene polymer (F) produced by polymerizing alkylene oxide in an initiator by making a composite metal cyanide complex (E) into a catalyst. An organic polymer (A) whose total amount of an ionic impurity it has a hydroxyl silicon group expressed with a formula (1), and is 50 ppm or less.

$$-R^2-SiX_2R^1-3-a \cdots (1)$$

As for R², a hydroxyl group or a hydroxyl basis, and a of substitution of the carbon numbers 1-20 or an unsubstituted univalent hydrocarbon group; and R¹ are the integers of 1-3 a divalent organic group

and X among a formula

[Claim 21] The room-temperature-curing nature constituent according to claim 1 whose organic amine compound (C) is a compound which has with a carbon numbers of 20 or less amine, or a hydroxyl silicon group expressed with an amino group and a formula (2) to intramolecular.

$$-R^2-SiX_2R^1-3-a \cdots (2)$$

As for a divalent organic group and R¹ a hydroxyl group or a hydroxyl basis, and b of substitution of the carbon numbers 1-20 or an unsubstituted univalent hydrocarbon group, and X¹ are [R²] the integers of 1-3 among a formula.

[Claim 31] The room-temperature-curing nature constituent according to claim 1 or 2 which is an ionic impurity in which an ionic impurity contains metallic compounds and/or an alkali metal compound resulting from a composite metal cyanide complex (E).

[Claim 41] An organic polymer (A) an ionic impurity contained in a polymer (F). After an ionic impurity contained in a polymer (F) by removing this salt from a polymer (F), after considering it to a polymer (F) as an insoluble salt atomically shall be 50 ppm or less. The room-temperature-curing nature constituent according to any one of claims 1 to 10 which is an organic polymer produced by introducing a hydroxyl silicon group into a polymer (F).

[Claim 51] An organic polymer (A) an ionic impurity contained in a terminal unsaturated group introduction thing (G) of a polymer (F). After an ionic impurity contained in a terminal unsaturated group introduction thing (G) by removing this salt from a terminal unsaturated group introduction thing (G), after considering it to a terminal unsaturated group introduction thing (G) as an insoluble salt atomically shall be 50 ppm or less. The room-temperature-curing nature constituent recording to any one of claims 1 to 3 which is an organic polymer obtained by making it react to a silicon hydride compound expressed with a terminal unsaturated group introduction thing (G) and a formula (3).

$$HSiX_2R^1-3-a \cdots (3)$$

A hydroxyl group or a hydroxyl basis, and a of R¹ are the integers of 1-3 among a formula A substitution of the carbon numbers 1-20 or an unsaturated univalent hydrocarbon group, and X.

[Claim 6] The room-temperature-curing nature constituent according to any one of claims 1 to 5 whose hydroxyl value conversion molecular weights of a polymer (F) are 5000-30000.

[Translation done.]